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ATC Report No. 00-121



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Effect of Coating Cure
Time on Adhesion and
Explosion Avoidance

Concrete Scoping Tests

Demonstration of
Explosion Prevention with
Non-Condensable Gas
Injection

Final Report to the
Sponsor Companies

David D. León
Ray T. Richter
Ingot & Solidification Platform

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Packaging Coating and Surface Technology



Creating Value through Technology

ALCOA INC.

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DISCLAIMER

This report is limited both in purpose and scope and is intended simply to summarize data obtained through a limited testing program conducted by Alcoa, Inc., under the financial sponsorship of the Aluminum Association, Inc. This study was solely designed to characterize the performance of a select number of protective coatings that can reduce the potential of molten metal explosions. It does not purport to address all situations which may arise under production conditions. No attempt has been made in this report by the Aluminum Association, its member companies or Alcoa to formulate recommendations or draw any conclusions concerning the relative explosion avoidance of the four protective coatings studied. Accordingly, neither the Aluminum Association, its member companies nor Alcoa makes any warranty, expressed or implied, or assumes any responsibility or liability, whether based on warranty, contract, negligence, strict liability, product liability, or otherwise with respect to use of the data herein.

EXECUTIVE SUMMARY

During the period of 1995 August through 1997 March, research contracted by the Aluminum Association on behalf of a group of sponsoring companies, identified three alternate coating materials which would be an acceptable replacement for Porter International's 7001 (Tarsol Standard). These coatings were:

1. Intertuf 132HS a coal tar epoxy by Courtaulds
2. Multi-Gard 955CP a 100% solid epoxy by Carboline
3. WiseChem E-115 a 100% solid epoxy by ESP

As a result of the research performed by Alcoa Inc. and Oak Ridge National Labs (ORNL), new questions were raised:

1. Can lower cure times than those designated by the coating vendors be used without compromising their explosion protection?
2. Are the alternate coatings as protective when applied on concrete surfaces?
3. Can the injection of non-condensable gases into the water serve as an alternate to the use of protective coatings?

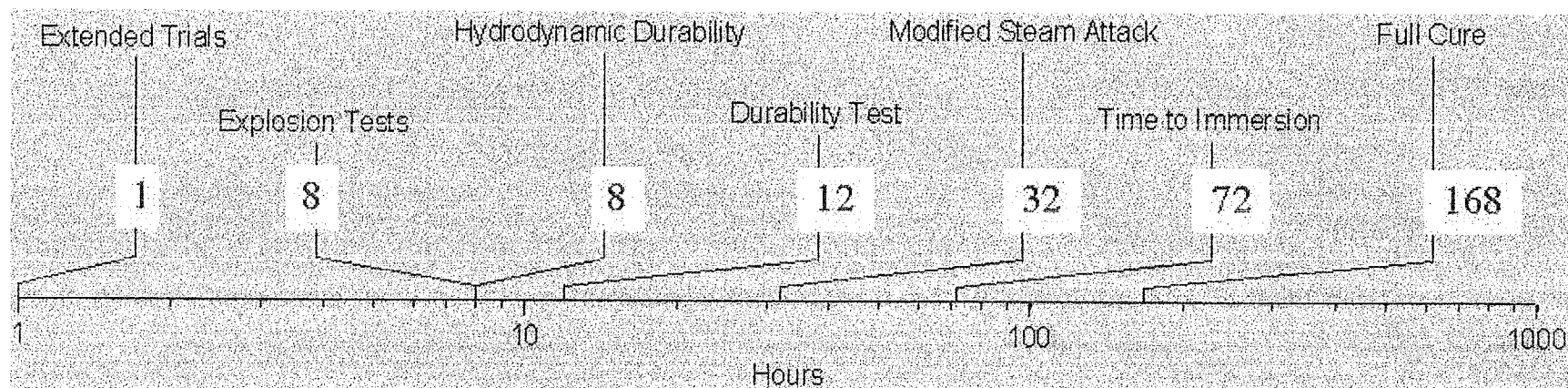
A multi-step approach was developed combining the expertise developed at Alcoa and Oak Ridge National Labs. The three coatings which emerged from the previous program, plus WiseChem E-212-F, were characterized using Differential Scanning Calorimetry, a Modified Steam Attack test, various Durability techniques, Oak Ridge's SETS equipment and the ATC Explosion Bunker.

The limited testing program showed that all the coatings can prevent explosions in casting pits, with acceptable adhesion performance at curing times below the original vendor recommendations and much lower than Full Cure (168 hr.). This reduced cure time was different for each coating. The Sponsors are reminded to take into consideration the coating location and pit conditions that the coating will be exposed to prior to deciding upon an In-Service time below Full Cure. The figures following this summary consolidates all the results of this program in graphical form.

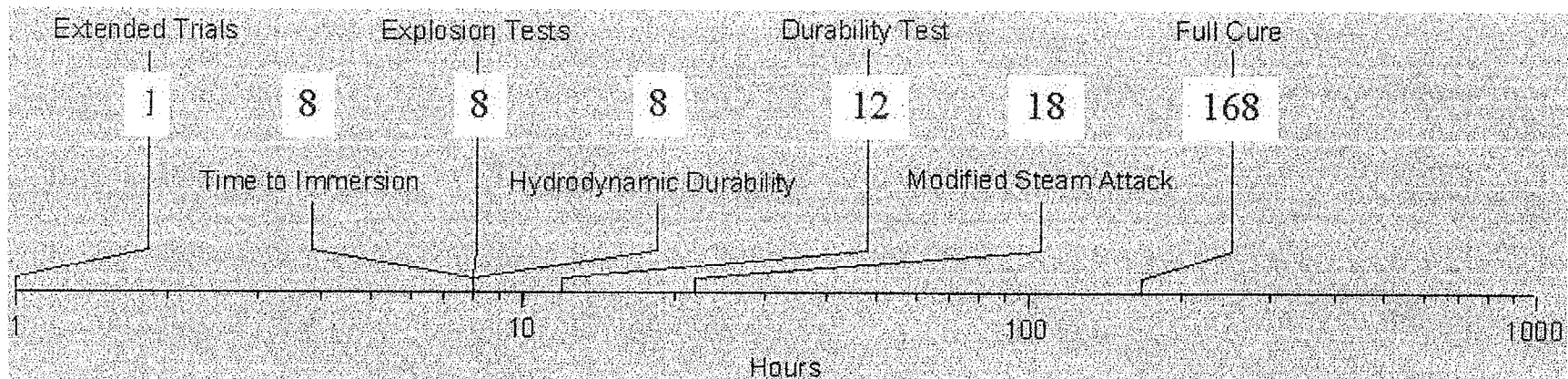
Unfortunately, a control test could not be developed for molten metal explosion testing on concrete containers. Durability testing did show that the adhesion performance of these coatings, at the vendor's recommended cure time, was similar to that of steel containers.

Although the use of Non-Condensable Gas Injection to prevent explosions was demonstrated in the laboratory using ORNL's apparatus, this success did not manifest itself in the Standard Molten Metal Explosion Test. Further investigation of the differences between the ORNL SETS apparatus and the 50 lb. molten metal test may be warranted.

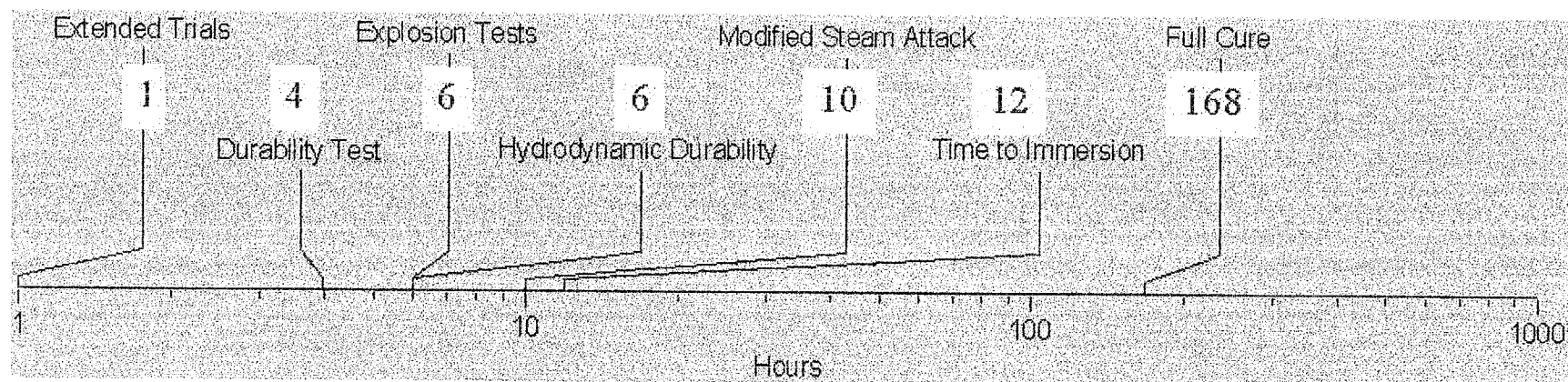
Summary Cure Time Results for Intertuf 132 HS



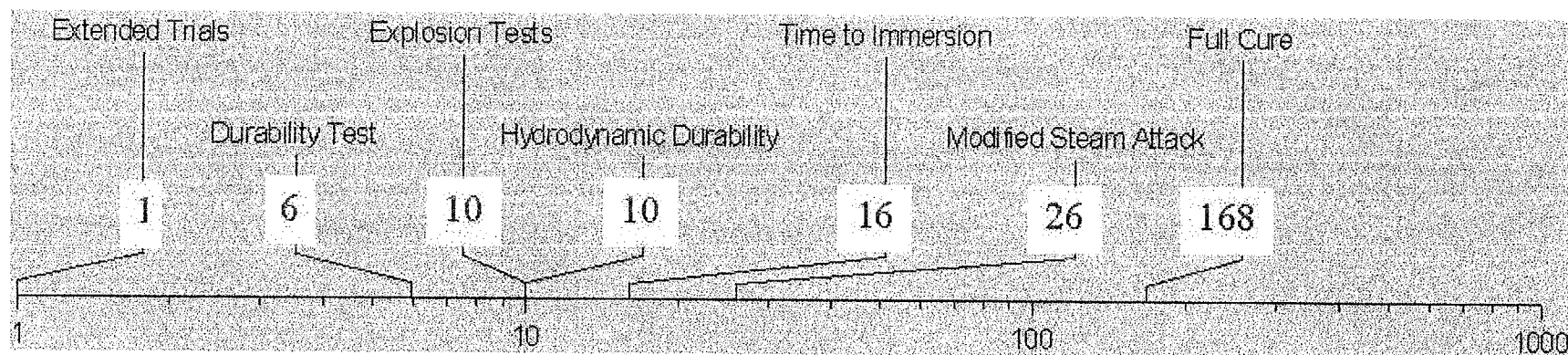
Summary Cure Time Results for Multi-Gard 955 CP

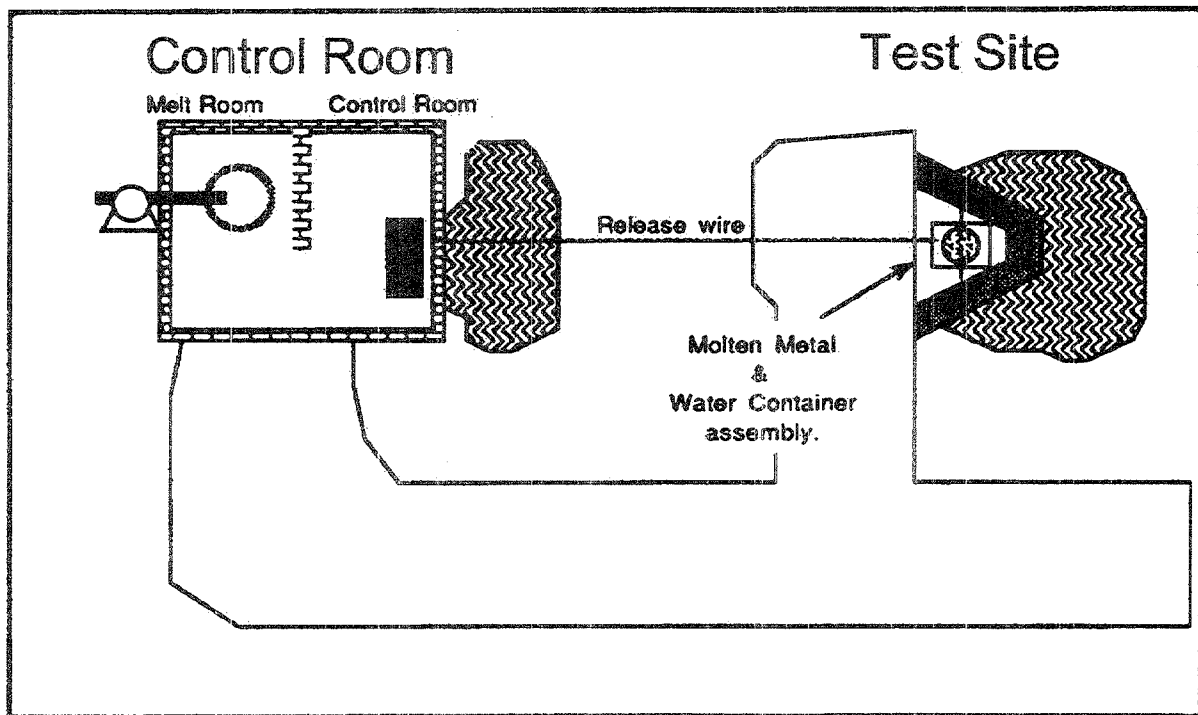


Summary Cure Time Results for WiseChem E-115



Summary Cure Time Results for WiseChem E-212-F





Alcoa Technical Center Explosion Bunker

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I. Background

The casting of molten metal to produce ingot is one of the most common practices performed in the Aluminum Industry. In this environment, there will always be the risk of a sudden release of molten aluminum onto the casting equipment, pit walls or pit bottom, due to process upsets. Since water is used as the main quenching media, during any molten metal spill there is the potential for a molten aluminum-water explosion to occur. Data collected by the Aluminum Association [1] shows that between 1980 and 1995, there were a total of 1190 incidents reported industry-wide, 423 (35.5%) occurred during casting operations. These incidents resulted in 40 fatalities, of which 10 (25%) occurred during casting operations. There is clearly a need to continue improving the safety of aluminum casting operations.

During the period of 1995 August through 1997 March, research contracted by the Aluminum Association on behalf of a group of sponsoring companies, identified three alternate coating materials which would be an acceptable replacement for Porter International's 7001 (Tarsel Standard) [2]. Alcoa identified these coatings through a series of selection criteria including: 1) An industry-standard molten metal explosion test, 2) A multiple-exposure test to measure durability, and 3) An external shock impact test. Sketches showing the equipment used at the ATC Explosion Bunker for these tests have been included in Attachment I. The final three coatings selected by the research team and the Sponsor Companies were:

1. Intertuf 132HS, a coal tar epoxy by Courtaulds
2. Multi-Gard 955CP, a 100% solids epoxy by Carboline
3. WiseChem E-115, a 100% solids epoxy by ESP

Two issues arose during this investigation:

1. Given the long cure times recommended by the manufacturer for the best coating candidates, what is the effect of reduced cure or water immersion times on coating adhesion and their effectiveness in preventing molten metal/water explosions?
2. For the new coatings tested, what is the smallest uncoated area on the pan bottom which would still initiate an explosion?

Following the review of this work, two additional issues were forwarded:

1. Oak Ridge National Labs proposed an alternate technique to prevent molten aluminum / water steam explosions via the injection of non-condensable gases. This technique needed to be validated using the established 50 lb drop test.
2. Although all three of the alternate coatings are rated for steel and concrete use, none were evaluated for explosion avoidance on concrete surfaces. Could a control and a series of explosion tests be devised to evaluate this?

In response to the request by the sponsors of the previous contract, Alcoa formulated a program to address the Cure time, Concrete and Non-condensable gas issues. A multi-step approach was developed. Alcoa and vendor coating expertise was combined with research related to steam explosion prevention at Oak Ridge National Labs (ORNL) under a Cooperative Research And Development Agreement (CRADA) with the Aluminum Association.

WiseChem E-212-F, which had been tested in the previous study, and has been used in production over the last 20 years, was also included in the characterization studies for comparison.

Alcoa was awarded Contract No. 422 in August of 1998. The companies sponsoring this research included:

Alcan International

Alcoa Inc.

Carboline Company

Century Aluminum Co.

Columbia Falls Aluminum

Comalco Research & Technology

Commonwealth Aluminum

E.S.P., Inc.

Kaiser Aluminum & Chemical

Logan Aluminum

Norandal, Inc.

Norsk Hydro Aluminum

A.P. Pechiney

Southwire, Inc. – NSA

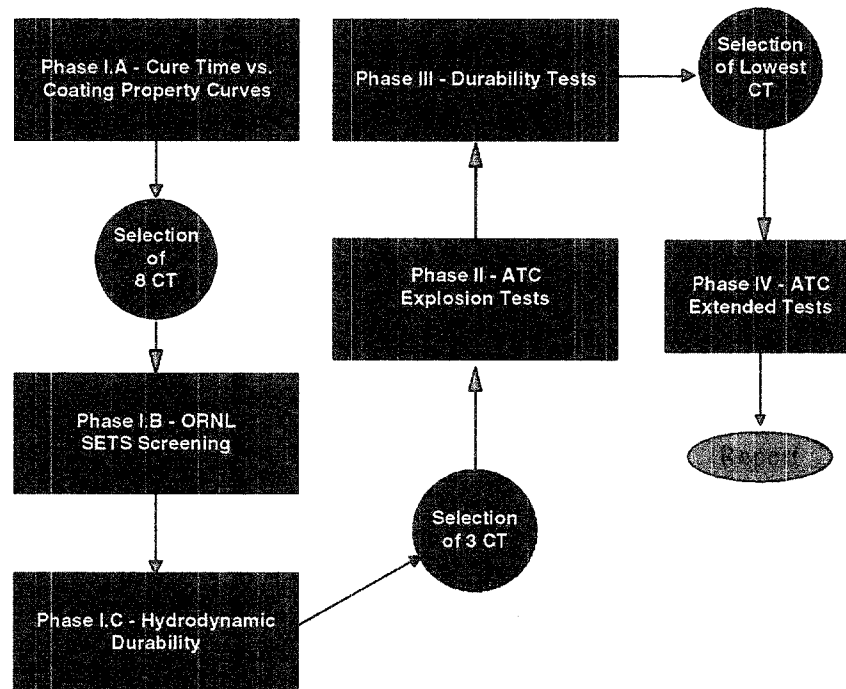
VAW of America

Wagstaff, Inc.

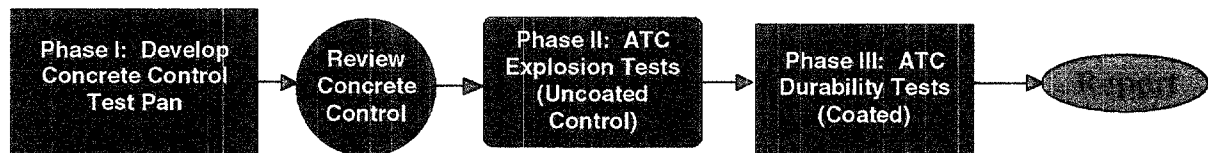
II. Experimental Procedure

The three-part contract was further subdivided in several tasks as shown in Figure 1. Table 1 shows the proposed test matrix for this program.

Effect of Cure Time:



Concrete Scoping Tests:



Non-Condensable Gas Injection Demonstration:

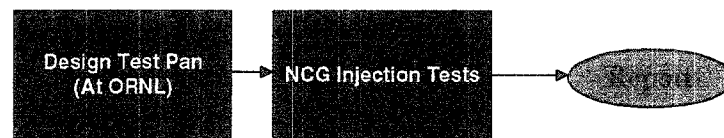


Figure 1: Aluminum Association Contract No 422 Program Flow

Table 1: Proposed Test Matrix for each Phase

Phase	Location	No. of Cure Times	Total No.** of Tests
Effect of Cure Time:			
I.A = Develop Coating Characteristic vs. Cure Time Curves	ATC	up to 22	166
I.B = Verification of selected cure times at Oak Ridge National Labs	ORNL	8	48
I.C = Hydrodynamic Durability Tests	ATC	8	##
II = Verification trials at the Explosion Bunker	ATC	3	60 + 5
III = Durability Tests	ATC & ORNL	2	32 - ATC # - ORNL
IV = Extended Tests	ATC	1	60 + 5
Evaluation on Concrete Surfaces:			
I. = Development of a control test pan for concrete surfaces	ATC	1+	n/a
II. = Explosion tests on control (uncoated) pans	ATC	1	5X X=1,2,3 or 4
III. = Durability tests on coated concrete pans	ATC	1	20 - 5X Pending Phase II
Effect of deliberate non-condensable gas injection:			
I. = Design and construction of test pans	ORNL	n/a	n/a
II = Validation tests	ATC	1*	5

- ORNL would run multiple exposures until the shock/vibration spectrum indicates a loss of protection.

- Four panels would be exposed simultaneously during one drop, for a total of four drops.

* - Refers to number of process conditions to be tested. Based on ORNL recommendations.

** - Assumes WiseChem E-212-F will be part of the evaluation matrix.

+ - The vendor's recommended cure time will be used.

A. Effect of Cure Time

All of the coatings tested under the Aluminum Association Contract No. 343: *Investigation of Coatings which Prevent Molten Aluminum/Water Explosions*, were evaluated for explosion avoidance using as a minimum the recommended cure times as provided by the manufacturer. (See Table 2.) The main issue among the Sponsor Companies involved the potential reduction in casting productivity caused by these long cure times. Other issues included the various definitions used by the vendors, the methodology used to determine "cure times," and the effect of time on water immersion and coating adhesion.

Table 2: Cure Times for the Candidate Coatings

Coating	Time to Touch	Time to Immersion	Recommended In-Service Time **	Full Cure
Intertuf 132 HS	6 hr. @ 75°F	72 hr. @ 77°F	168 hr. @ 75°F	168 hr. @ 75°F
Multi-Gard 955CP	8 hr. @ 75°F	8 hr. @ 75°F	168 hr. @ 75°F [#]	168 hr. @ 75°F
WiseChem E-115	6 hr. @ 77°F	12 hr. @ 70°F	12 hr. @ 70°F	168 hr. @ 70°F
WiseChem E-212-F	6 hr. @ 77°F	16 hr. @ 70°F	16 hr. @ 70°F	168 hr. @ 70°F
<i>Tarset Standard *</i>	<i>3 hr. @ 75°F</i>	<i>72 hr. @ 75°F</i>	<i>168 hr. @ 75°F</i>	<i>168 hr. @ 75°F</i>

* Shown for comparison only. This coating is no longer available.

** Each vendor defined minimum "in-service" time differently. For Intertuf and Multi-Gard it was defined as full cure, and for WiseChem it was time to immersion.

[#] Carboline's original In-Service time recommendation. This was later changed to 8 hr. in June of 2000 based on this program.

The only way to insure that the proper reduced in service time is used for the various coatings is by developing Cure Time vs. Explosibility data. As noted in Figure 1, a four-phase program was developed to measure the effect of reducing cure time.

Phase I.A = Develop Coating Characteristic vs. Cure Time Curves

Two analytical techniques, typically used in the coatings industry, were used to measure the changes occurring in the selected coatings over time. These techniques were:

- Differential Scanning Calorimetry (DSC) — A technique which measures energy changes in the coating as it is heated at various rates. This tool is used to predict chemical rates of reaction. It is also the primary tool that will be used to predict the coating's cure cycle.
- Modified Steam Attack Test — A procedure used for evaluating the adhesion of a coating. Steam under pressure is blown over the coating to simulate the hot environment within casting operations.

The DSC kinetics analysis showed that curing of these coatings appear to be governed by two separate processes: 1. Drying, and 2. Cross-linking (or cure). In all cases, the endotherms occurring during solvent evaporation dominated any exotherms making the analysis more difficult.

Borchardt and Daniels' kinetic theory (B-D) was used as a screening tool with corrections for filler content as determined by Thermo-Gravimetric Analysis. Table 3 shows the results of the B-D kinetics analysis.

Table 3: Borchardt and Daniels' Kinetics¹

Coating	n	E _a (kJ/mole)	Log Z (min ⁻¹)	ΔH (J/g)	Std Error (sec ⁻¹)
Intertuf 132HS	2.44	62.4	8.10	106.6	0.0833
WiseChem E-115	1.26	64.9	8.96	300.2	0.0028
Multi-Gard 955CP	1.01	60.3	7.62	320.8	0.0412
WiseChem E-212-F	2.46	71.1	9.04	198.2	0.0150

where n = reaction order
E_a = Activation energy
Z = pre-exponential factor
ΔH = Heat of reaction

The above information was used to develop predictive Conversion Curves for the various coatings. Note from above that the WiseChem E-115 and the Multi-Gard 955CP are first order reactions with very well behaved kinetics. The higher order reactions measured on the Intertuf 132HS and the WiseChem E-212-F make these more difficult to predict. Figures 2 through 5 show the Conversion Curves for the four coatings. These curves provide information on how fast the various coatings cure at select temperatures.

¹ Taken at 5°C/minute heat-up rate.

The Modified Steam Attack Test was performed by the Cleaning and Coatings Group at ATC. The coatings and panels were prepared following the same guidelines used in the first program. In order to "pass" the Mod. Steam Attack Test, the panel must undergo a five psi

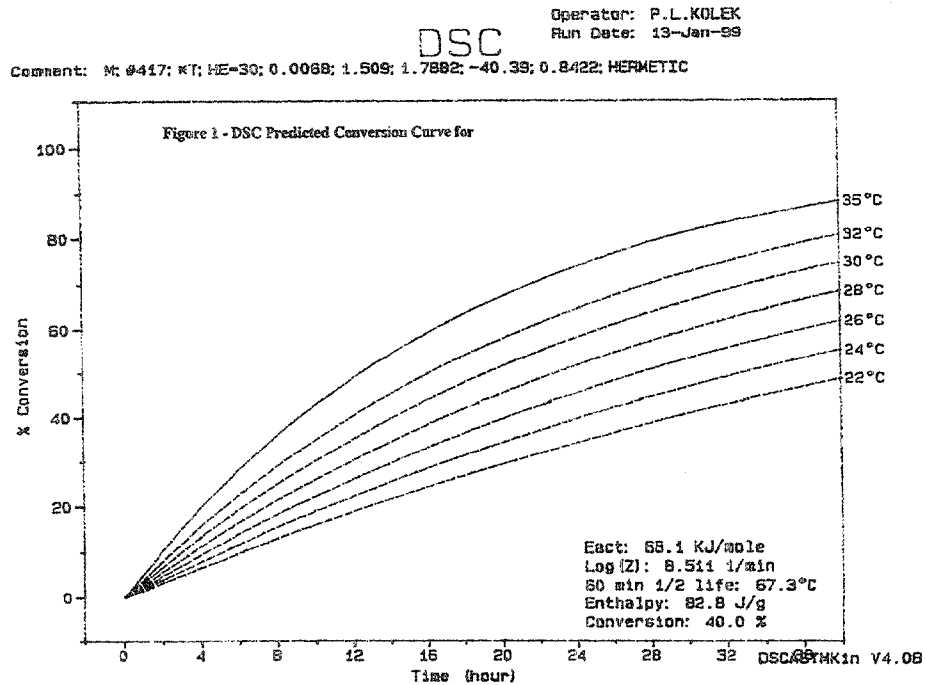


Figure 2 – Conversion curve for Intertuf 132 HS

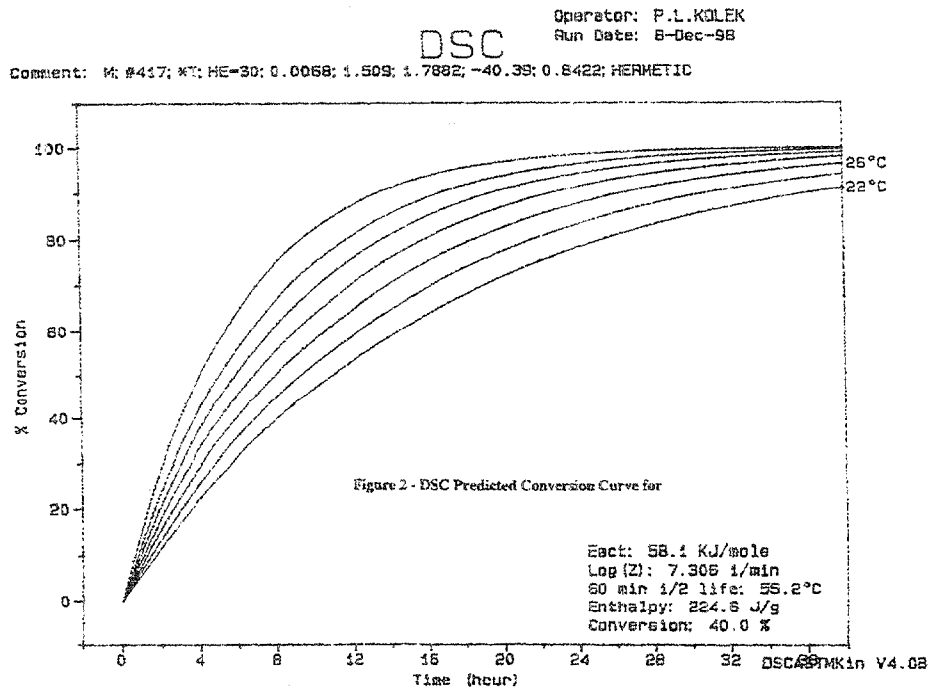


Figure 3 – Conversion curve for Multi-Gard 955CP

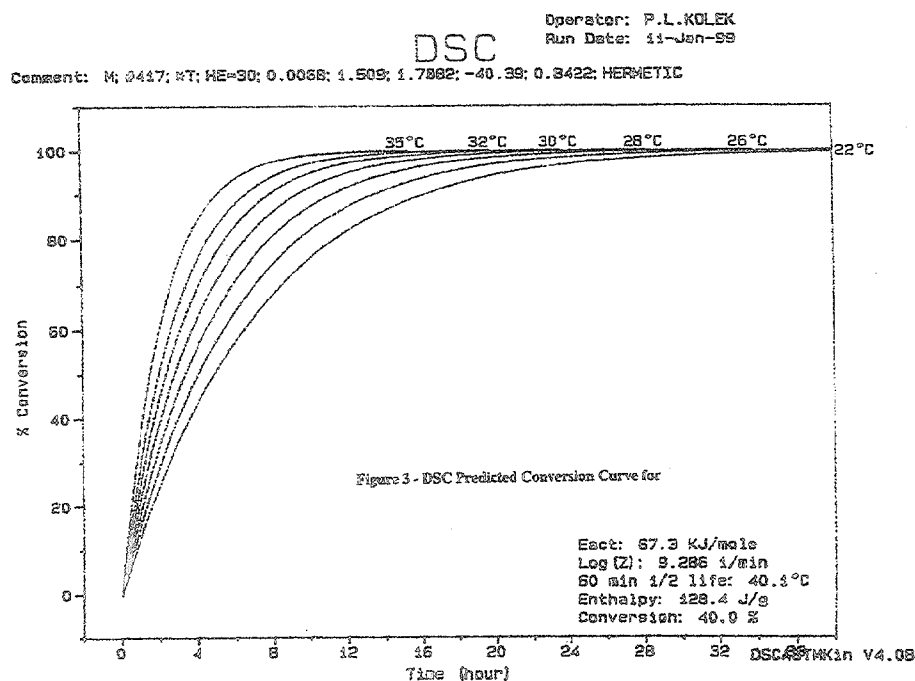


Figure 4 - Conversion curve for WiseChem E-115

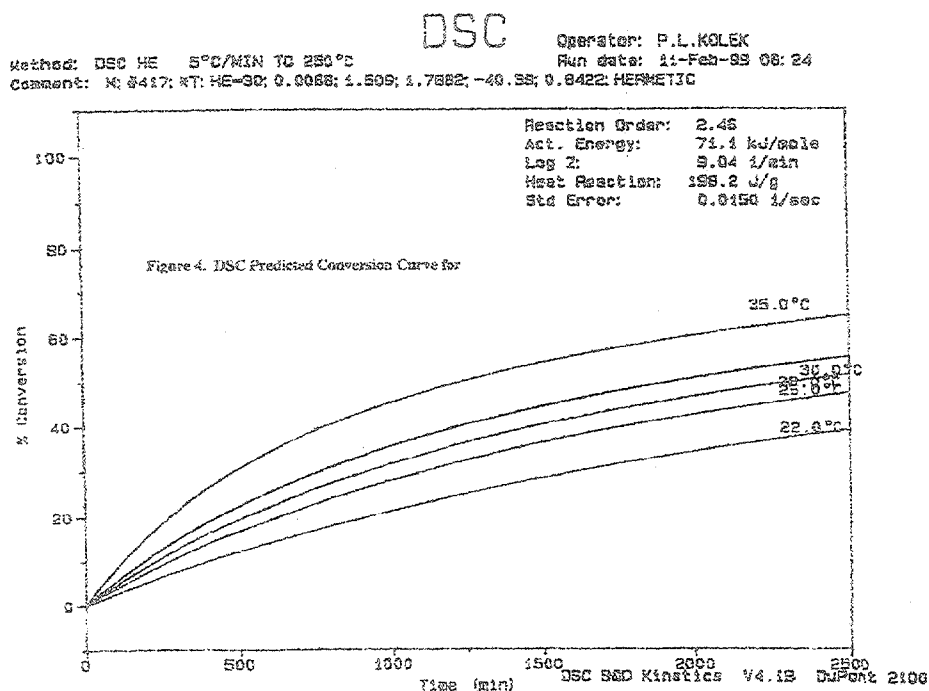


Figure 5 - Conversion curve for WiseChem E-212-F

steam attack without deformation of the coating or other loss of adhesion. Table 4 shows the results of the modified steam attack test.

Table 4: Modified Steam Attack Test Results

Coating	Cure Time (hr)								
	4	8	10	16	18	22	24	26	32
Intertuf 132HS	Liquid/ Tacky	Liquid/ Tacky	==	==	==	Tacky	Failed Steam	==	Passed Steam*
Multi-Gard 955CP (w/ additional data)	Liquid/ Tacky	Tacky	==	Failed Steam	Passed Steam	Passed Steam	==	==	==
WiseChem E-115	Liquid/ Tacky	Partial Fail **	Passed Steam	==	==	==	==	==	==
WiseChem E-212-F (second test)	Liquid/ Tacky	==	==	==	==	Failed Steam ***	==	Passed Steam	==

== Indicates coating not tested at this cure time.

* The samples may have passed the steam test sooner.

** This sample failed at 6 hours (not shown). The failure at 8 hours is less severe than at 6.

*** Material was dry to the touch even though it failed the test.

Phase I.B = Verification of selected cure times at Oak Ridge National Labs

The DSC and Modified Steam Attack data from Phase I.A provided information for selecting eight cure times to be evaluated by Oak Ridge National Labs on their Steam Explosion Triggering Studies (SETS) equipment under the Cooperative Research and Development Agreement (CRADA). See Table 5. The ORNL results have been reported separately [3].

Table 5: Cure Time Selection for Phases IB and IC

COATING	CURE TIMES (hr)
Intertuf 132HS	2, 3, 6, 8, 20, 24, 28, 32
Multi-Gard 955CP	2, 3, 6, 8, 12, 16, 20, 22
WiseChem E-115	2, 3, 6, 8, 10
WiseChem E-212-F	2, 3, 6, 8, 10, 12, 14

The Modified Steam Attack Tests provided the basis for the upper bound of cure times selected. The lowest cure times were based on typical casting pit turnaround times once coated, and the time-to-touch as defined by the manufacturers.

Attachment II contains a table which compares the cure times selected above with the amount (%) of curing as predicted by the Conversion Curves.

Phase I.C = Hydrodynamic Durability Tests

Simultaneously with ORNL, ATC performed hydrodynamic durability tests at its research casting pit. Test panels were prepared and exposed to the casting water environment by placing them on the platen of ATC's Advanced Development Casting Pit during a cast. The test was designed to evaluate the effect of direct water impingement on coating adhesion at the various cure times. Panels were characterized pre- and post-exposure to determine differences.

Coated panels with a target cure time were placed underneath the bottom block on the bottom block base, commonly called the "dog-house," of ATC's Research Casting Pit [4]. For the test, 8 x 16 inch "V"-shaped steel panels were used, one coating per side. The panel followed the 20-degree angle of dog house. The panels were exposed to the casting pit environment, including direct impingement of water from the mold after cooling the ingot, for full length (180 in.) casts. Water flow rates from the mold were approximately 1.3 gpm/in. Figure 6 shows the panel placement.

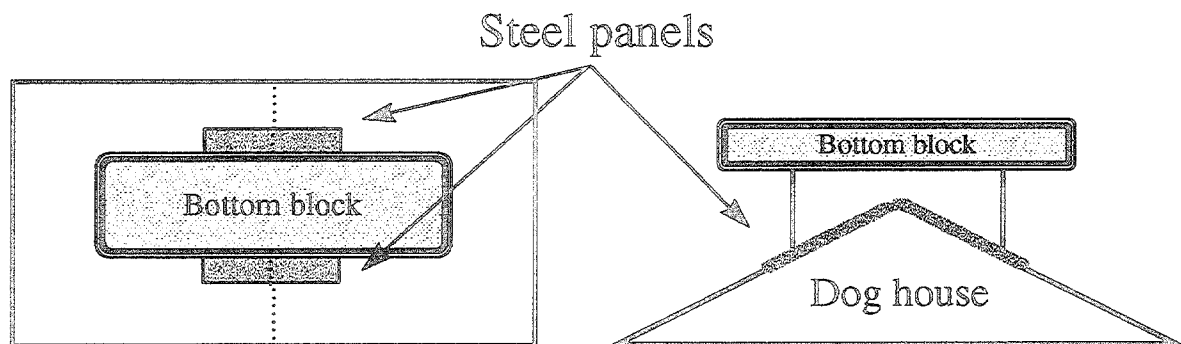


Figure 6: Hydrodynamic Durability Test Set-up

After the cast the panels were graded as: 1) pass, 2) fail or 3) borderline based upon the coating adherence to the panel. Table 6 contains the results of the Hydrodynamic Durability Tests.

Table 6: Hydrodynamic Durability Test Results

WiseChem E-115		
Cure time (hr)	Pass/Fail/Borderline	
2	B	
2.5	B	
3	B	
6	P	
8	P	
10	P	

WiseChem E-212-F		
Cure time (hr)	Pass/Fail/Borderline	
2	F	
3	B	
3.5	F/B	
6	F & B	
8	B	
10	P	
12	P	

Multi-Gard 955 CP		
Cure time (hr)	Pass/Fail/Borderline	
3	F	
3.5	F	
6	B	
8	P	
12	P	
16	P	
20	P	
22	P	

Intertuf 132 HS		
Cure time (hr)	Pass/Fail/Borderline	
3	F	
3.5	F	
6	F	
8	P	
12	P	
20	P	
24	P	
28	P	
32	P	

P = Pass

B = Borderline

F = Fail

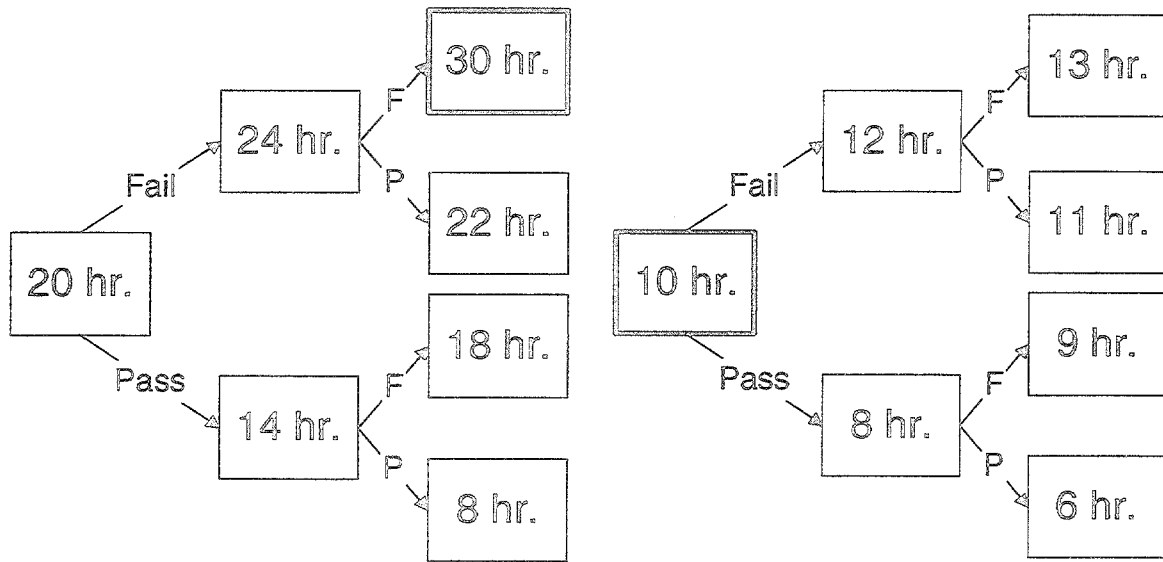
F&B = Two tests, each different result.

F/B = Most of the panel was borderline except for small sections which failed.

Attachment I contains select pictures of the panels after exposure to the casting environment. Shown are samples of the pass/fail/borderline ranking system used by Alcoa.

Phase II = Verification Trials at the ATC Explosion Bunker

ORNL's SETS test results were combined with the Alcoa results from Phases I.A and I.C to help identify cure-times to be tested in Phase II. Alcoa's goal through these tests was to identify the lowest In-Service cure time for each coating. A pass/fail decision tree was established for explosion testing of the coatings at different cure times. (Figures 7a and 7b show the pass/fail tree for Multi-Gard 955 CP and WiseChem E-115 respectively.) Under this concept, "Pass" was defined as being protective against explosions for all five repeat tests at the same cure time.



a. Multi-Gard 955CP

b. WiseChem E-115

Figure 7: Decision Tree for Selection of In-Service Times
for Molten Aluminum/Water Explosion Trials

The lowest cure time of each decision tree corresponded to the minimum cure time at which the coatings passed the Hydrodynamic Durability Test. The in-service times noted in the bold boxes denote the results from the SETS trials at ORNL. They represent the minimum cure time needed in order to avoid explosive triggering shocks.

Pending results from ORNL's Phase I.B, Alcoa performed explosion tests on Intertuf 132HS and WiseChem E-212-F. The starting point was selected as the minimum in-service time at which the individual coatings would pass the Hydrodynamic Durability Test. Table 7 summarizes the results for all four coatings.

Table 7 – Results of Molten Aluminum/Water Explosion Trials for all Coatings

Coating	Cure Time(s) Tested (hr)	Number of Explosions out of Number of Tests
Intertuf 132HS	8	0 out of 5
Multi-Gard 955CP	20	0 out of 5
	14	0 out of 5
	8	0 out of 5
WiseChem E-115	10	0 out of 5
	8	0 out of 5
	6	0 out of 5
WiseChem E-212-F	10	0 out of 5

Phase III = Durability Tests

Multiple molten aluminum exposures to a coated, submerged substrate was the primary method of measuring durability. Based on previous Sponsor Company comments, the coatings were characterized between tests. Surface condition and coating thickness were two of the main criteria used to evaluate the coatings after repeated exposure to molten aluminum.

Alcoa performed some preliminary molten metal durability tests at the lowest in-service time tested in the previous phase for each of the candidate coatings. Table 8 shows the results of the tests.

Table 8 – Results of Molten Metal Durability Tests

Coating	In-service Time	Test Number at which Failed	Test Number at which Failed at Full Cure*
Intertuf 132 HS	8 hr.	2	3
Multi-Gard 955 CP	8 hr.	2	4
WiseChem E-115	6 hr.	5	3
WiseChem E-212-F	10 hr.	3	4

* These results were obtained during the previous contract work and have been included for comparison purposes.

Following these tests, the Sponsor Companies approved a modification to the goals of Phase III and IV. Originally it was planned to identify a single short cure time for each coating which would have good adhesion characteristics and have good results in the durability

explosion tests, however the revised program was targeted at identifying two different short cure times:

Objective 1. What is the minimum in-service cure time required to minimize the potential of molten metal/water explosions AND provide durability?

Objective 2. What is the absolute minimum in-service cure time required which reduces the risk of molten metal/water explosions, taking into account direct and indirect casting water attack? Implies that, in a production environment, the coating MUST be recoated if an upset condition (i.e., a molten metal spill) would occur.

Durability testing continued until an in-service cure time was found where the coating survived two or more tests. The results of this phase are shown in Table 9.

Table 9 – Molten Metal Durability Test Results

Coating	In-service Time	Test Number at which Failed
Intertuf 132 HS	8 hr.	2
Intertuf 132 HS	12 hr.	3
Multi-Gard 955 CP	8 hr.	2
Multi-Gard 955 CP	12 hr.	3
WiseChem E-115	2 hr.	2
WiseChem E-115	4 hr.	4
WiseChem E-115	6 hr.	5
WiseChem E-212-F	3 hr.	2
WiseChem E-212-F	6 hr.	6
WiseChem E-212-F	10 hr.	3

Phase IV = Extended Tests

In this phase, 15 standard molten aluminum/water explosion tests were performed on all four coatings at the one hour cure time. The extended tests provide additional statistical confidence that explosions may be prevented at this short cure time. In addressing Objective 2, one hour was selected as the cure time for the standard explosion test performed on each coating. “Pass” was defined as being protective against explosions for all repeat tests at the same cure time.

All coatings completed this testing successfully with no explosion occurring in any of tests as shown in Table 10.

Table 10 - Results of Extended Tests
(All Tests performed at 1 hr. Cure Time)

Coating	# of failures out of # of tests
Multi-Gard 955 CP	0 out of 15
WiseChem E-115	0 out of 15
WiseChem E-212-F	0 out of 15
Control (un-coated)	6 out of 6

B. Concrete Scoping Tests

During the review meetings between the Alcoa Research Team, the Aluminum Association and the Sponsoring Companies, the issue of explosion avoidance on coated concrete surfaces was discussed. Although all three of the alternate coatings are recommended by the manufacturers for use on both steel and concrete substrates, only steel pans were evaluated during the previous project.

There were several issues to consider:

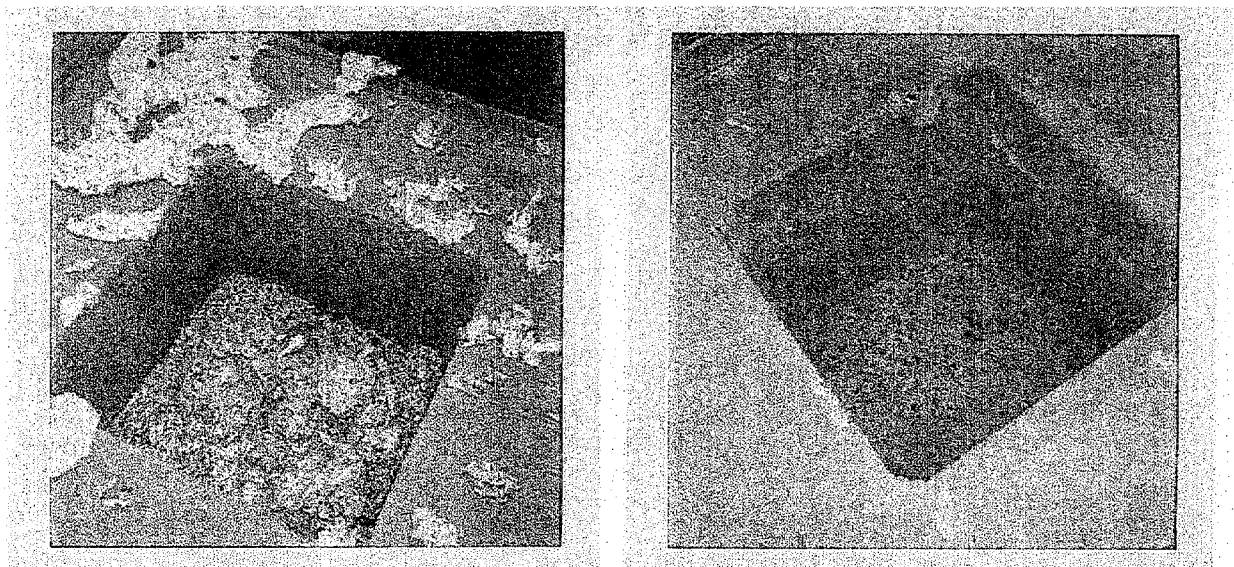
- The industry does not have a standardized method for testing concrete containers.
- Historical data by Hess and Brondyke was inconclusive regarding tests with concrete substrates. There were a very limited number of tests performed.
- The controls used in the previous program are based on our ability to obtain an explosion when the pans are specifically prepared to do so.
- A procedure would have to be devised so that tests on uncoated cement would result in an explosion.

The first task under this project was to survey select Sponsors to determine the various concrete specifications used by each company. Specifications were obtained from Alcoa Inc., Hydro Aluminum, Pechiney and Reynolds Metals.

In general, most specifications for cast-in-place concrete are based on minimum design strengths ranging from 3,000 to 4,000 psi., referring to the appropriate location Building Codes along with American Concrete Institute (ACI) and American Society for Testing

Materials (ASTM) standards. These standards provide for guidelines in use of cement, additives, aggregate (sand/gravel) and water in the mix. In addition, standards were provided for surface flatness and preparation.

With this information, ATC designed the test containers to be evaluated. The Alcoa containers have an inside dimension similar to the one used in the previous program on steel (i.e., 12 x 12 x 12 inch as cast), with six in. thick walls all around. (See Figure 8.) This configuration was selected to meet the strength criteria and essentially simulate the construction of a casting pit. The containers were designed to 3,500 psi. The first two containers Alcoa tested were in the as-cast condition (rough surface, with isolated pores).



(a)

(b)

Figure 8: Alcoa Concrete Pans

(a) Pan shown after exposure to molten metal. Walls are six in. thick. (b) Pan after removal of the frozen metal. Note attack on the bottom of the container.

During the program, Alcoa performed an assortment of tests with the concrete pans to find an appropriate control. Figure 9 shows a photograph of the ATC Explosion Bunker with the concrete container in place.

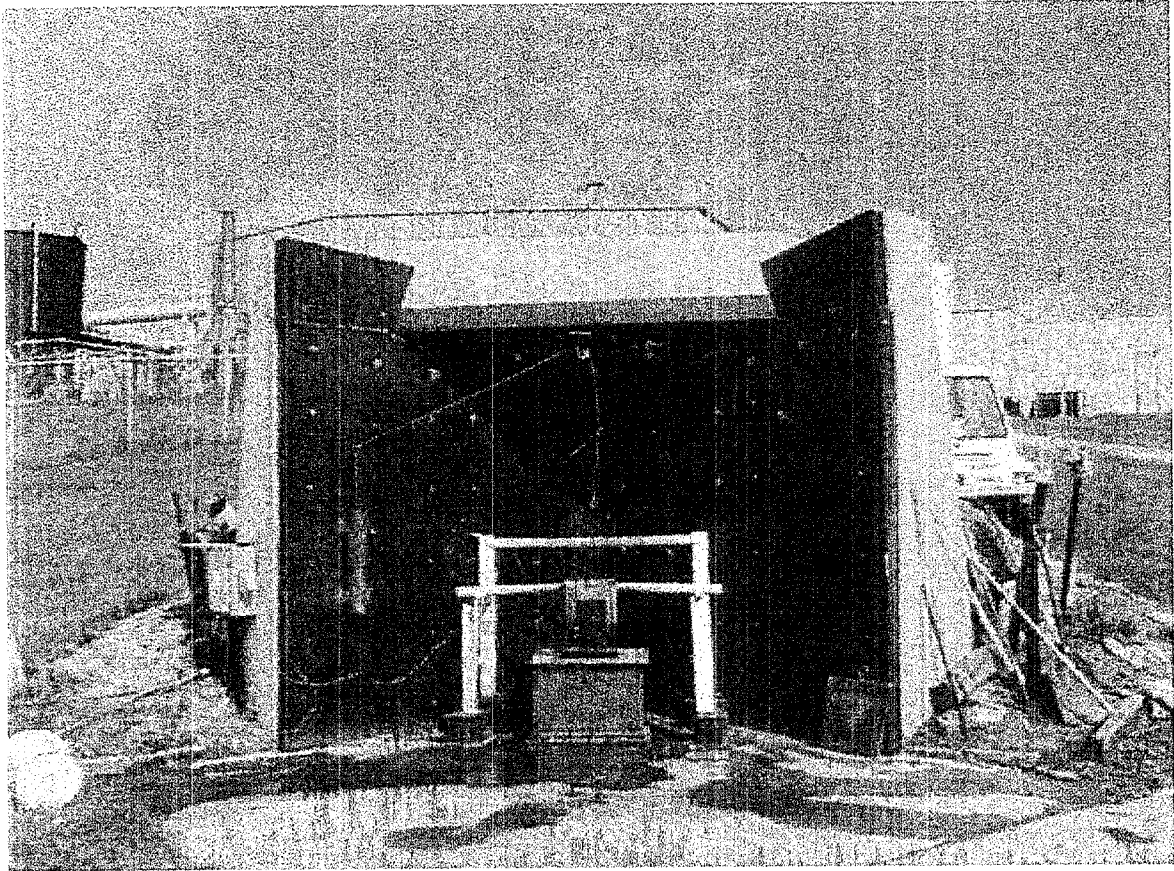


Figure 9: Alcoa's Explosion Bunker Test Site

Following are the results from the various tests performed at ATC:

Tests run with 3,500 psi strength concrete:

- | | |
|--|-----------------------------|
| • With thin, rough cement coating ² | 0 explosions out of 3 tests |
| • As above, chipped to show aggregate | 0 explosions out of 1 test |
| • Cement coated, with 1 inch water | 0 explosions out of 1 test |
| • As cast | 0 explosions out of 1 test |

² Two tests ran with metal below 1400°F

Tests run with cold water following ORNL's SETS discovery:

- Stone polished³ 1 explosion out of 2 tests

Tests run with ORNL recipe for high strength concrete:

- As cast³ 0 explosions out of 2 tests
- Sand-blasted³ 0 explosions out of 2 tests

Since no control could be established, the Research Team discontinued searching for a control. The Sponsors agreed to use the Durability Test method to measure coating degradation and adhesion, following Hess' example [5, 6].

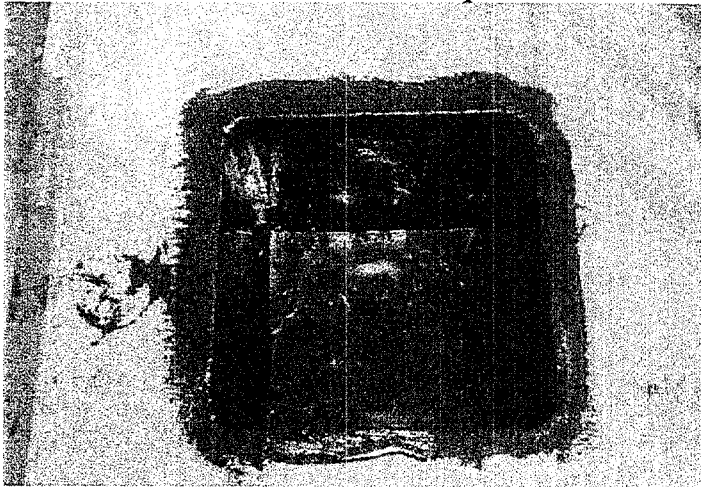
The following procedures were followed:

1. The standard 50 lb. melt drop test (including $\leq 50^{\circ}\text{F}$ water) was performed on the original concrete containers. These were the containers using a concrete specification for an average casting pit.
2. Surface preparation included whip-blasting the surface, patching or buttering any holes in the surface, and a final whip-blast prior to coating.
3. One container was prepared for each of the four coatings.
4. We used the current set of "in-service" cure times recommended by the manufacturer.
5. After each test we performed a visual check of the container and documented adhesion and bare spot issues.
6. Three tests were performed per container.

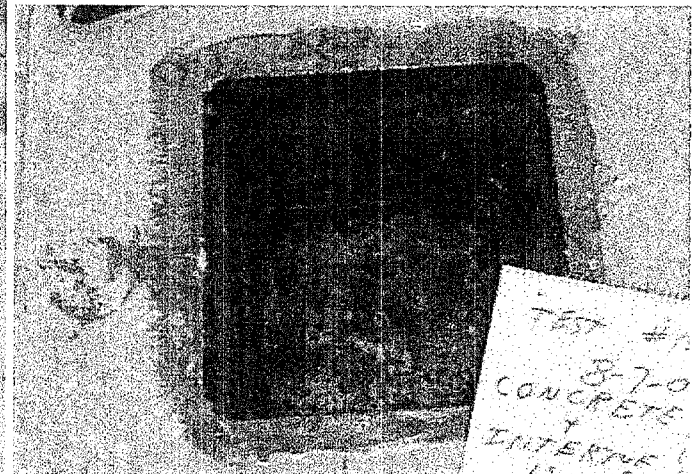
Following are a series of photographs showing the concrete pans before and after three exposures to molten metal. Erosion of the coating was noted after every exposure to molten metal. The amount of erosion varied with each coating.

³ $< 45^{\circ}\text{F}$ water.

Before Molten Metal Exposure

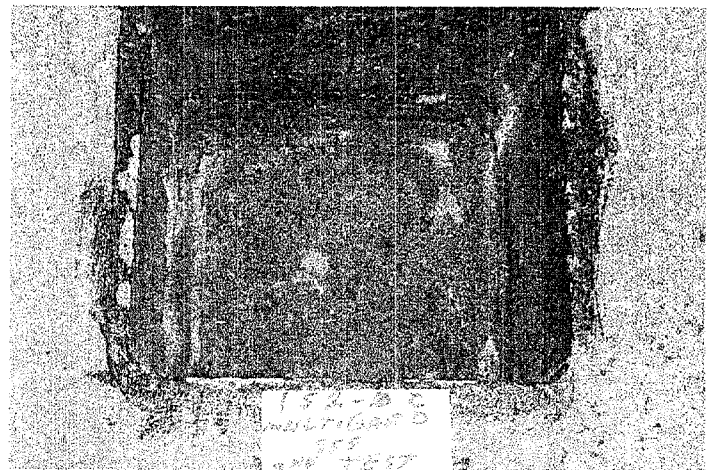
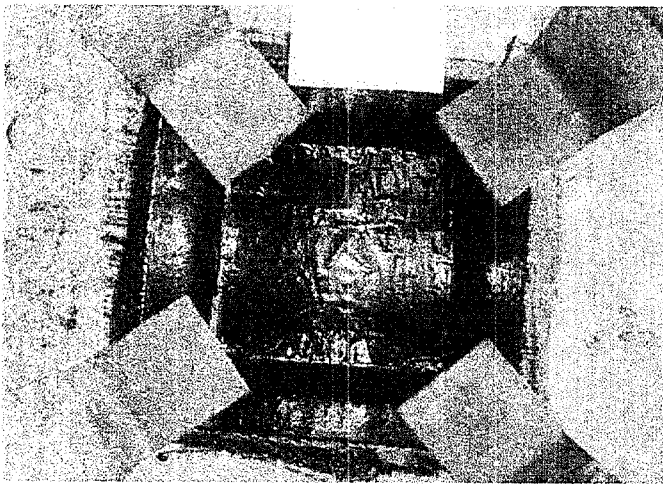


After Three Exposures to Molten Metal



Intertuf 132 HS observations:

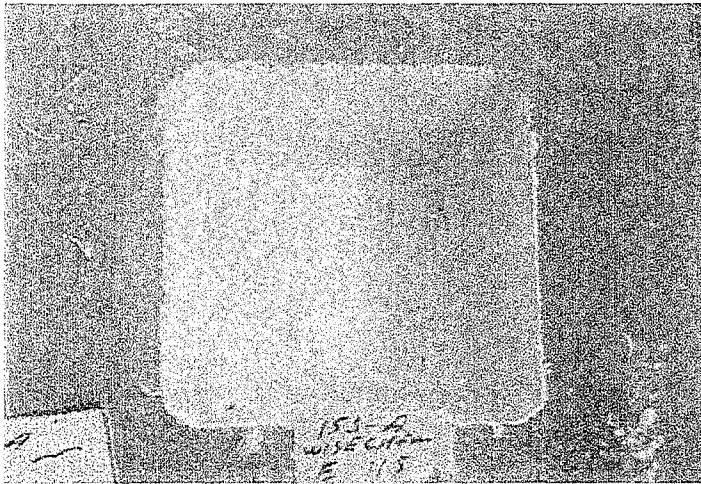
There was no loss of adhesion in any part of the container. There were several areas on the bottom and sides where the coating was eroded (note lighter areas in photo above), but not to bare concrete.



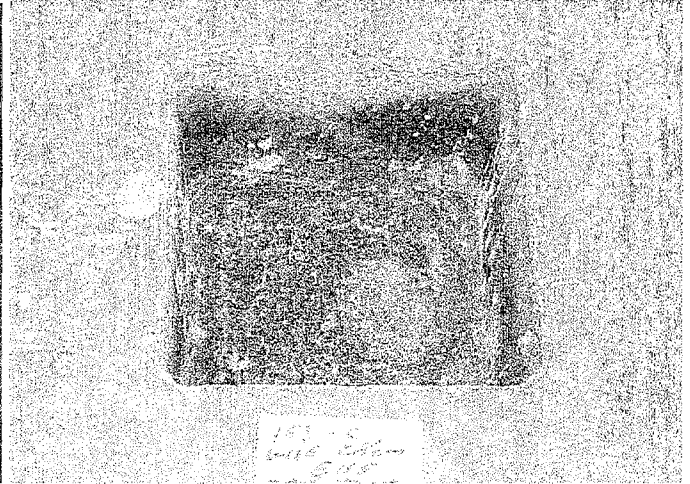
Multi-Gard 955CP observations:

There was several areas with loss of adhesion, only on the bottom of the container (note non-coated areas in photo above). Individual spots were less than one in² in area.

Before Molten Metal Exposure

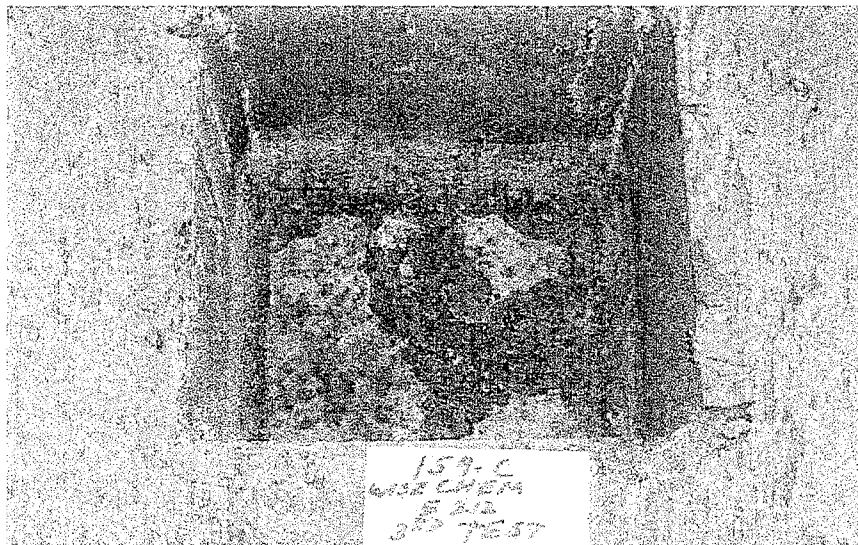


After Three Exposures to Molten Metal



WiseChem E-115 observations:

There was no loss of adhesion in any part of the container (the coating loss in the upper and lower area noted in the right photo was caused during removal of the aluminum from the previous test). There were no areas on the bottom and sides where the coating eroded to bare concrete.



WiseChem E-212-F observations:

There was no loss of adhesion in any part of the container. There were several areas on the bottom and sides where the coating was eroded (note lighter areas in photo above), but not to bare concrete. This coating tends to expand when heated by the molten metal. Material erosion occurs, therefore, in layers. The top layer erodes or was removed with the aluminum from the previous test. Note the charred dark areas vs. the lighter coated ones.

C. Non-Condensable Gas Injection Demonstration

As a result of extensive testing conducted at ORNL, evidence evolved which indicates that the injection of non-condensable gases (such as air) through a porous plate (over which molten metal is relocating) should provide for a very effective suppressant of steam explosions. This evidence has been derived via:

1. Actual testing with molten metal-water combinations in which molten metal (in small scale) when dropped in water would explode without air injection, but would be inert to explosions when air bubbles were injected through a porous plate.
2. SETS facility tests wherein air injection through a porous plate gives explosive boiling shock spectrum very similar to that obtained with surfaces coated with paints such as WiseChem, etc.
3. Tests with pyrolysis of paints which indicate that coatings which provide suppression release significant amounts of non-condensable gases upon thermal attack.

In order to confirm this hypothesis and validate the novel technique for prevention of explosions in the aluminum industry casting pits, Alcoa Inc. proposed that explosion tests be conducted at ATC. ORNL would design and fabricate the pans with a simple gas injection system and suitable instrumentation. ATC would be responsible for conducting five Standard Explosibility tests.

ATC prepared eight-inch diameter perforated plates in pre-oxidized condition. Two hole patterns were tested by ORNL, 0.5 mm perforations in a ½ inch and in a one inch square pattern. Based on Oak Ridge's recommendation, ATC built five 12 x 12 x 12 pans with 0.5 mm perforations in a one-inch square pattern. These were modified by ORNL into three container types as seen in Figure 10.

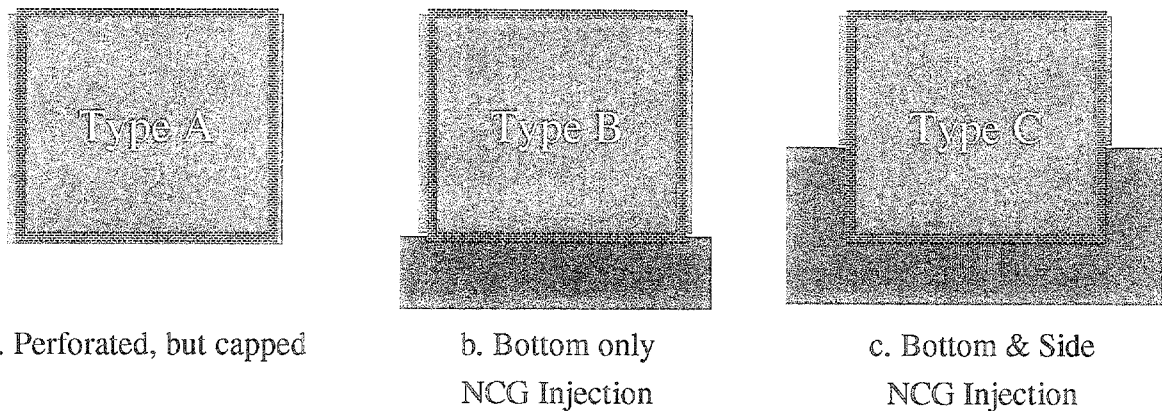


Figure 10: Oak Ridge National Lab's Non-Condensable Gas Injection Test Pans

Two tests were performed:

1. Type C Pan, Set-up at similar gas flow to SETS 2.9 cfm on sides, 3.5 cfm on bottom
2. Type B Pan, Set-up at minimum flow which provided bubbling action (1.4 cfm)

Both of these tests resulted in explosions. No further tests were scheduled after these results.

III. Conclusions

This report is intended simply to summarize data obtained through a limited testing program conducted by Alcoa Inc. under financial sponsorship of the Aluminum Association. It is important to understand that these tests may not represent conditions in all aluminum casting pits. Therefore, the explosion avoidance results from this program do not guarantee that the same results will occur under production casting conditions.

Based on the results obtained from this focused program, we provide the following conclusions:

- Differential Scanning Calorimetry was used to predict the curing cycle of these coatings. The initial intent was to use these DSC Conversion Curves to select curing times where physical changes were detected. The analysis showed that all of the coatings have smooth curing cycles so this method could not be used to make selections.
- All four coatings survived the Modified Steam Attack at times less than the vendor recommended In Service cure time. Although this provides a screening tool for the cure times, it cannot predict performance in the casting pit.

- Under the conditions tested, all four coatings can withstand direct water impingement at times less than the vendor recommended In Service cure time. This minimum cure time will be different for each coating and will also depend upon the exact pit operating conditions.
- All coatings can be washed away at short enough cure times. Because of this, it will be critical to take into consideration the coating location and pit conditions that the coating will be exposed to prior to deciding upon a minimum cure time for each coating.
- Based upon the explosion durability tests at In Service cure times less than vendor recommended, all four coatings produced results comparable to the durability tests at full cure times. The minimum cure time to provide similar results was different for each coating.
- Using the industry standard explosion test, no explosions occurred with any of the four coatings at cure times below full cure even down to one hour of curing. All coatings avoided an explosion in every one of the 15 tests performed with one hour curing.
- All four coatings achieved acceptable explosion durability at less than 40% curing as predicted by the DSC Conversion Curves.
- A control test could not be developed for molten metal explosion testing of concrete containers. This is consistent with previous Alcoa Inc. work which showed that explosions were possible, but not predictable.
- Although the use of Non-Condensable Gas Injection to prevent explosions was demonstrated in the laboratory using ORNL's apparatus, this success did not manifest itself in the Standard Molten Metal Explosion Test. Further investigation of the differences between the ORNL SETS apparatus and the 50 lb. molten metal test may be warranted.

IV. References

- [1] Epstein, S. G., *Update on Molten Aluminum Incident Reporting*, Light Metals 1997, Proceedings of the 126 TMS Annual Meeting, Orlando, FL, February 1997.
- [2] León, D. D., Richter, R. T., Levendusky, T. L., *Investigation of coatings which prevent molten aluminum/water explosions - Final report to the Sponsor Companies*, Report No. 97-475-18-DDL, 1997 June.
- [3] Taleyarkhan, R. P., et al., *Fundamental Studies on Molten Aluminum-Water Explosion Prevention in Direct-Chill Casting Pits*, Light Metals, 2001, J. L. Anjier, Editor, pp. 793-795.

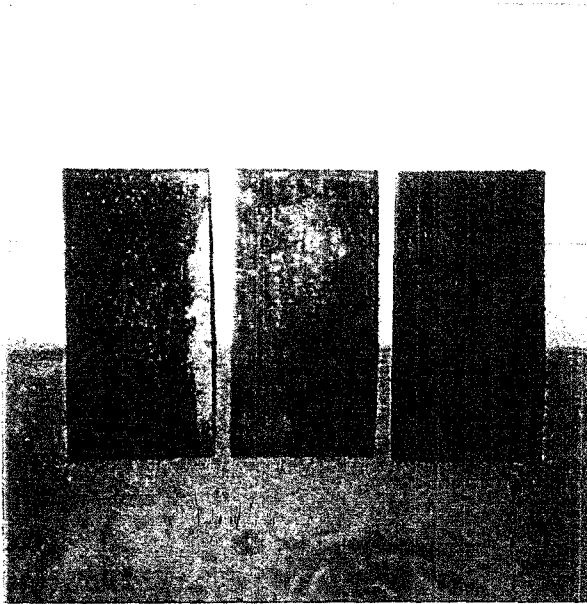
- [4] Richter, R. T., Adomaitis, P. R., Hildeman, G. J., *Real-Time Video Monitoring of Ingot Casting*, Light Metals 1996, Wayne Hale, Editor, pp. 941-948
- [5] Hess, P. D. and Brondyke, K. J., *Study of Explosions of Molten Aluminum in Water*, Alcoa Research Laboratories Report No. 7-68-BE21, October 1968.
- [6] Hess, P. D., Miller, R. E., Wahnsiedler, W. E., and Cochran, C. N., *Molten Aluminum/Water Explosions - 1979*, Alcoa Research Laboratories Report, October 1979.

V. Contributions and Acknowledgments

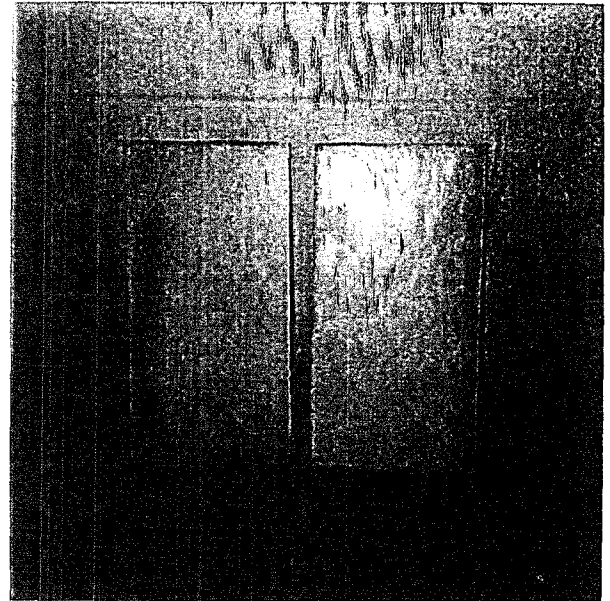
The authors would like to recognize the contributions of Mr. John Jacoby (Consultant), the Dept. of Energy's Off. of Industrial Technology (supporters of ORNL's work), William Straub and John Hartill (lead operators of the ATC Explosion Bunker), and to the all of the Aluminum Association's Sponsor Companies.

ATTACHMENT I

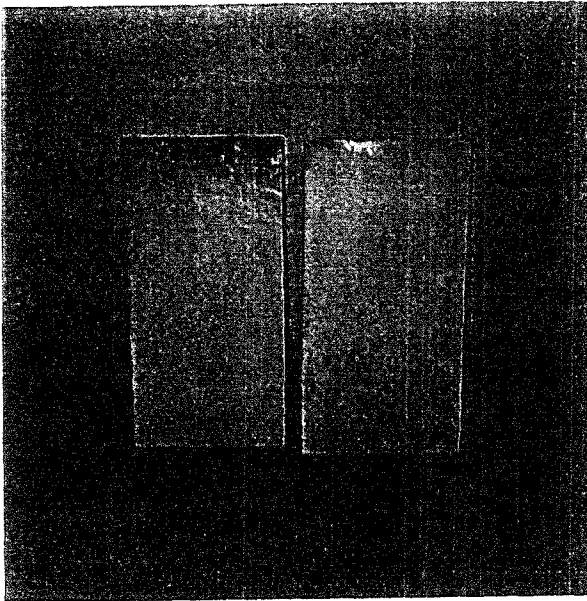
HYDRODYNAMIC DURABILITY TEST – SAMPLE PANELS



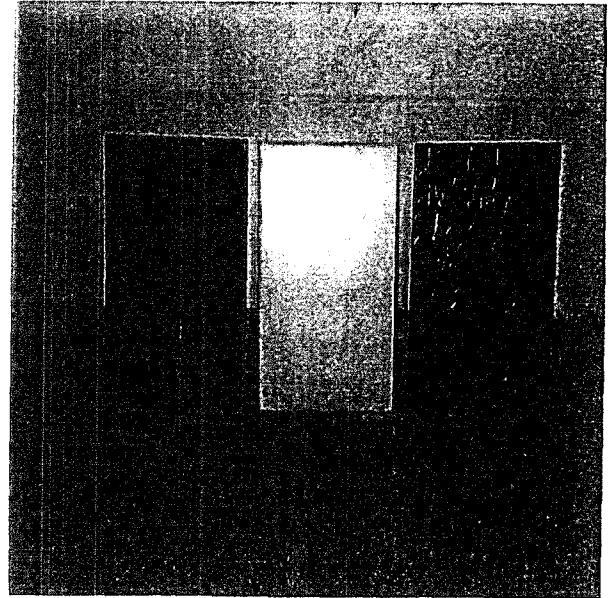
Examples of "Fail" - Coating eroded completely to bare steel or extremely thin coating left behind.



Examples of "Fail - to - Borderline" - Erosion noted in the plane of the water fall only.



WiseChem E-115 and E-212-F - Note how material tore off on right panel instead of eroding.



Examples of "Pass" - coating remains intact or just slightly pushed.

ATTACHMENT II

PHASE I A CURE TIME SELECTIONS WITH CONVERSION PREDICTIONS:

COATING									
Intertuf 132 HS	Cure time	2	3	6	8	20	24	28	32
	Pred. % Conversion	3	5	9	13	28	33	36	42
Multi-Gard 955CP	Cure time	2	3	6	8	12	16	20	22
	Pred. % Conversion	11	17	32	39	53	63	71	75
WiseChem E-115	Cure time	2	3	6	8				10
	Pred. % Conversion	24	36	58	68				76
WiseChem E-212-F	Cure time	2	3	6	8		10	12	14
	Pred. % Conversion	3	18	32	40		48	55	58